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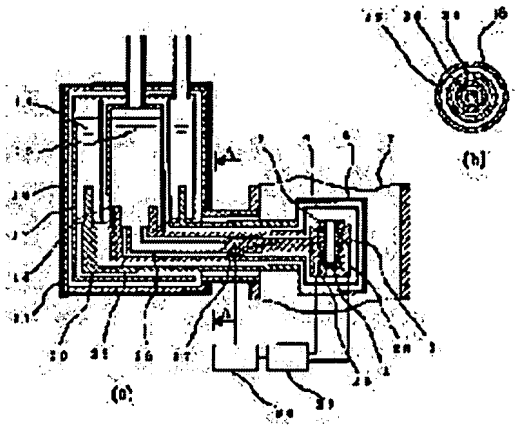
(54) SAMPLE COOLING DEVICE

(57)Abstract:

PROBLEM TO BE SOLVED: To compensate generation of extremely low temperature and a temperature in a sample cooling device for an electron microscope or the like.



SOLUTION: A sample cooling device is formed as a refrigerant storage vessel 16 of two-tank structure provided with a liquid helium storage vessel 18 in the inside of a liquid nitrogen storage vessel 17, independent two first/ second heat transfer members 23, 26 connected to the liquid helium storage vessel 18 and a third heat transfer member 20 connected to the liquid nitrogen storage vessel 17 are each almost cylindrically included in the outside of the first heat transfer member 23, heat transfer and heat shield are both provided. The first heat transfer member 23 is formed hollow, so as to allow a refrigerant to flow in, also heat transfer area is made variable by a piezoelectric element 27. In a sample 1 or sample holder 2, or a cooling member 3 loading the sample 1 or sample holder 2, a heater 25 and a

temperature sensor 28 are provided, a temperature control device 29 and a piezoelectric element driving gear 30 are controlled by a closed loop, so as to be set controlled at an arbitrary temperature, the temperature is compensated.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the very-low-temperature sample cooling system used combining the equipment which installs a sample in vacuum housings, such as an electron microscope.
[0002]
[Description of the Prior Art] As very-low-temperature sample cooling systems, such as the conventional electron microscope, the equipment in which sample cooling is possible to 5K or less very-low-temperature field is in JP,63-32847,A like a publication.
[0003] Drawing 1 shows the configuration of very-low-temperature sample cooling systems, such as the conventional electron microscope.
[0004] Install the sample stage 6 which consisted of the second [which encloses the first cooling member 3 and the first cooling member 3 which carry out heat contact with a sample 1 or the sample holder 2, and which are cooled to very low temperature approximately cylindrical, and performs a heat-shield], and third cooling members 4 and 5 in the electron microscope mirror body 7, and are mutually-independent out of the electron microscope mirror body 7. The first liquid helium reservoir container 8 which cools said first cooling member 3, the second liquid helium reservoir container 9 which cools the second [which performs a heat-shield], and third cooling members 4 and 5, and the liquid nitrogen reservoir container 10 are installed. It was combined with the first cooling section 3 and second, and third cooling member 4 and 5 by the first heat-conduction member 11 and second, and third heat-conduction member 12 and 13, respectively, and the first and second liquid helium reservoir containers 8 and 9 and the liquid nitrogen reservoir container 10 enabled cooling of a sample to very low temperature. Thus, the heating value which flows into the low-temperature section by heat conduction and thermal radiation in the conventional very-low-temperature sample cooling system The liquid nitrogen cooling member 5 cooled by about 100 K between the sample 1 and the sample electrode holder 2, and the room temperature part since it was so few that the temperature of the elevated-temperature section which contacts or faces it was low is made to intervene. 100K or more liquid nitrogen cooling members 5 and the liquid HEUMU cooling member 3 of several K have the temperature gradient which is about 100 degrees. Furthermore, in order [contact or in order to make it not face], The second liquid HEUMU cooling member 4 is made to intervene between the liquid HEUMU cooling members 3 and the liquid nitrogen cooling members 5 which cool a sample 1 and the sample electrode holder 2. It considered as the Mie heat-shield structure surrounding a sample 1 and the sample electrode holder 2, and had become the structure which cools the three above-mentioned cooling members through three heat-conduction members, respectively from three refrigerant reservoir containers. However, by this method, when a liquid nitrogen reservoir container sets change of the heating value which flows into the low-temperature section by heat conduction and thermal radiation by one tub and a liquid helium reservoir container becoming required two tubs, and sample temperature as arbitration according to the heat input of a heater etc., the latent heat of vaporization of liquid helium is as small as 1/10 compared with liquid nitrogen, and the consumption of liquid helium is greatly

influenced by the slight heat flow rate close. From this, by 2 tub methods, the conventional liquid helium reservoir container will change with experiment conditions of sample cooling of the liquid helium consumption of each reservoir container, and will be determined by the liquid helium of the side by which the time amount (sample cooling experiment) which can be sample cooled is consumed early of the liquid helium of two tubs. This cannot perform effective use of expensive liquid helium, but will consume liquid helium to a large quantity, and lacks also in profitability. Moreover, a reservoir container ON **** activity becomes again about liquid helium, and workability is also missing. In order that the manufacturing cost of a reservoir container might also become three reservoir containers and a refrigerant container might naturally enlarge it, there were troubles, like for a reason, a manufacture price goes up. Furthermore, aerial vibration, such as an acoustic wave received from the oscillation and refrigerant container outside surface by evaporation of a refrigerant, is transmitted to a sample or a sample holder through a heat-conduction member, and the engine performance of an electron microscope etc. was reduced.

[0005]

[Problem(s) to be Solved by the Invention] In the very-low-temperature sample cooling system of the above-mentioned conventional technique, when cooling attainment temperature is determined by the cooling property of a refrigerant and a very-low-temperature sample cooling system, and it carries out setting-out control to an elevated-temperature side at the temperature of arbitration, and a heater etc. performs a heat input to a sample, a sample electrode holder, or the heat-conduction member to load, evaporation of a refrigerant will be brought forward and the sample cooling holding time's being shortened inevitably or an expensive refrigerant will be consumed to a large quantity. For this reason, it is necessary to reduce consumption of a refrigerant and to aim at improvement in profitability. Moreover, the liquid helium which the consumption of the liquid helium of two reservoir containers changed with 1:10 and small slight heat flow rate close compared with liquid nitrogen, and the sample cooling holding time was restricted by consumption of one liquid helium of the reservoir container whose number is two, and filled up two reservoir containers with the latent heat of vaporization of liquid helium is not used effectively.

[0006] Furthermore, a liquid nitrogen reservoir container needs to shorten the working hours with which one tub and a liquid helium reservoir container fill up a refrigerant in order to take great time amount to fill up a reservoir container with a refrigerant, since it has refrigerant container composition of two tubs.

[0007]

[Means for Solving the Problem] This invention makes the refrigerant reservoir container of a very-low-temperature sample cooling system 2 tub structures of a liquid-nitrogen reservoir container and the liquid-helium reservoir container in which it was prepared by that inside, prepares the heat-conduction member combined by the liquid-helium reservoir container inside the heat-conduction member combined with the liquid-nitrogen reservoir container, makes the other end of the heat-conduction section combined with this liquid-helium reservoir container the two heat-conduction sections which divided or became independent to two, and makes the heat-conduction section three layer systems. Furthermore, adjustable or a heat-conduction member is divided for the heat-conduction area of the heat-conduction member which cools a sample or a sample holder in respect of heat conduction, the consumption of liquid helium is restricted by controlling division section heat-conduction face-to-face heat contact time, a heat input function and a temperature detection function are prepared in a sample and a sample electrode holder or said sample, and a sample electrode-holder loading cooling member, and carrying out control compensation of the temperature of a sample or a sample electrode holder is realized.

[0008] The inclusion configuration of the two heat-conduction members cooled by liquid helium inside the heat-conduction member cooled with liquid nitrogen is carried out at three layer systems, and a heat-shield layer is prepared and it carries out endoergic [of the heat which flows into an interlayer] by liquid helium. Furthermore, a heater and a thermo sensor are installed in the cooling member which cools a sample or a sample holder, adjustable or a heat-conduction member is divided for the heat-conduction area of a heat-conduction member in respect of heat conduction, setting-out control is carried

out to an elevated-temperature side at the temperature of arbitration by controlling division section heat-conduction face-to-face heat contact time, and temperature is compensated. This reduces the consumption of liquid helium.

[0009]

[Embodiment of the Invention] Hereafter, one example of this invention is explained using drawing.

[0010] Projection association of the heat-conduction member is carried out in the example of this invention at the refrigerant reservoir container of 2 tub structures, and, as for (b), drawing 2 (a) shows [the other end of the heat-conduction member combined with the refrigerant reservoir container by the side of low temperature] the A-A sectional view of the heat-conduction member of (a) for two block diagrams of the very-low-temperature sample cooling system which served both as division, heat conduction of three layers, and a heat-shield approximately cylindrical. A sample 1 or the sample holder 2 carries out heat contact with this cooling system, uses liquid nitrogen 14 and liquid helium 15 for very low temperature, and is cooled by heat conduction. The refrigerant reservoir container 16 prepared out of the electron microscope mirror body 7 forms the liquid helium reservoir container 18 inside the liquid nitrogen reservoir container 17, includes the liquid helium reservoir container 18 in a liquid nitrogen reservoir container by the heat-shield member which carried out heat contact, and it constitutes with the refrigerant reservoir container of 2 tub structures which carried out heat insulation support of the liquid nitrogen reservoir container 17 and the liquid helium reservoir container 18 respectively, and were fixed to the refrigerant reservoir container 16. The heat-shield of the first cooling member 3 which cools a sample 1 or the sample holder 2 is carried out approximately cylindrical by the second and third cooling members 4 and 5. The end of the first heat-conduction member 19 is combined with said liquid helium reservoir container 18, it is divided approximately cylindrical, and said first cooling member 3 **** is combined with the second cooling member 4, and this first heat-conduction member 19 is cooled. The second heat-conduction member 20 combined with the liquid nitrogen reservoir container 17 is approximately cylindrical, and it is formed in the outside of the first heat-conduction member 19, is combined with the third cooling member 5, and is combined with the second cooling member 4, and a heat-shield is performed with said first heat-conduction member 19. Thus, the volume of the liquid helium reservoir container 18 can be enlarged, without enlarging the volume of the refrigerant reservoir container 16 by constituting from 2 tub structures of the liquid nitrogen reservoir container 17 and the liquid helium reservoir container 18. furthermore, the external heat input which flows into a sample 1 or the sample electrode holder 2 by heat conduction or radiation is absorbed on a first stage story with the large (about 10 times of a liquid helium refrigerant) liquid nitrogen refrigerant 14 of the latent heat of vaporization -- having -- the boiling point of the liquid nitrogen refrigerant 14 -- a low temperature side -- the liquid helium refrigerant 15 -- therefore, it is absorbed. By dividing approximately cylindrical, the other end of the first heat-conduction member 19 which combined with the liquid helium reservoir container 17 the first cooling member 3 which cools a sample 1 or the sample holder 2, and the first cooling member 3 which performs a middle heat-shield from the above-mentioned thing The heat input which the first cooling member 3, the second cooling member 4 which performs a middle heat-shield, and the first heat-conduction member 19 receive can be taken charge of as the latent heat of vaporization of a liquid helium refrigerant, and a temperature gradient can be given to the first cooling member 3 and the second cooling member 4. Heat exchange with a refrigerant is made easy by projecting in each refrigerant container in a part of first heat-conduction member 19 combined with the liquid nitrogen reservoir container 17 and the liquid helium reservoir container 18, and second heat-conduction member 20. Moreover, aerial vibration, such as an acoustic wave which forms a sound deadener 21 in the outer wall of the refrigerant reservoir container 16, and is received from the oscillation and refrigerant container outside surface by evaporation of a refrigerant, is reduced, and an oscillating inflow in a sample is reduced.

[0011] the heat-conduction member by which drawing 3 (a) was combined with the refrigerant reservoir container by the side of the low temperature of said example in the example of this invention -- two -- it became independent -- it is approximately cylindrical, and constitutes and (b) shows [heat conduction of three layers, and a heat-shield] the A-A sectional view of the heat-conduction member of (a) for the

block diagram of a **** very-low-temperature sample cooling system. That is, in this example, without dividing the first heat-conduction member 19 combined with the liquid helium reservoir container 18 of the example of drawing 1 , the second heat-conduction member 23 is formed in the outside of the first heat-conduction member 22 which became independent respectively approximately cylindrical, and it combines with the liquid helium reservoir container 18. The heat transfer system of the first heat-conduction member 22 and the second heat-conduction member 23 can be separated by this, and a heat-shield property and a sample cooling attainment property can be improved.

[0012] (b) shows the A-A sectional view of the heat-conduction member of (a) for the block diagram of the very-low-temperature sample cooling system of the structure where the refrigerant with which drawing 4 (a) made hollow the heat-conduction member combined with the refrigerant reservoir container by the side of the low temperature of said example in the example of this invention, and the refrigerant reservoir container was filled up flows into the centrum of a heat-conduction member. In this example, the liquid helium refrigerant 15 with which made hollow the first heat-conduction member 22 combined with the liquid helium reservoir container 18 of the example of drawing 3 , and the liquid helium reservoir container 18 was filled up flows into the centrum of the first heat-conduction member 24, and it filled up with the liquid helium refrigerant 15. Even if the distance of the refrigerant reservoir container 16 and the sample 3 cooled increases and a heat-conduction member (20, 23, 24) becomes long by enlargement of a very-low-temperature sample cooling system by this, a sample cooling attainment property is not reduced. The gas which attached slight dip to the centrum of the first heat-conduction member 24 at this time, and the cooling refrigerant evaporated can be made easy to discharge.

[0013] In the example of drawing 4 , although the first heat-conduction member 24 was made hollow, the second and third heat-conduction member 20 and 23 can be similarly made hollow, and a heat-shield property and a sample cooling attainment property can be raised further.

[0014] (b) shows the A-A sectional view of the heat-conduction member of (a) for the block diagram of the very-low-temperature sample cooling system which drawing 5 (a) made heat-conduction area adjustable in the location of the arbitration of the heat-conduction member combined with the refrigerant reservoir container by the side of the low temperature of said example in the example of this invention, and made temperature control and temperature compensation possible. As shown in drawing, when setting-out control of the temperature of the cooled sample 1 or the sample electrode holder 2 is carried out according to the heat input of heater 25 grade to an elevated-temperature side at the temperature of arbitration, a cooling refrigerant is made to evaporate according to a heat input. Setting-out control of the temperature of the sample 1 cooled without dividing the first heat-conduction member 26, controlling the amount of heat transfer by making heat-conduction area of the division section adjustable, and evaporating a cooling refrigerant in order to reduce the consumption of a refrigerant, or the sample electrode holder 2 can be carried out to an elevated-temperature side at the temperature of arbitration. A piezo-electric element 27 is formed in the division section of the first heat-conduction member 26, a heater 25 and a thermo sensor 28 are further formed in the first cooling member 3 in which the sample 1 or the sample electrode holder 2 was carried, heat-conduction area of the division section of the first heat-conduction member 26 is made adjustable by controlling a temperature controller 29 and the piezo-electric element driving gear 30 by the closed loop, setting-out control of the temperature of a sample 1 or the sample electrode holder 2 is carried out at arbitration, and temperature is compensated with this example. It does not need to be based on a piezo-electric element 27, but, in the case of other driving means, it is necessary to take a driving means by which a heat input is not made the first heat-conduction member 26.

[0015] Drawing 6 (a) is divided in the location of the arbitration of the heat-conduction member combined with the refrigerant reservoir container by the side of the low temperature of said example in the example of this invention, division section heat-conduction face-to-face heat contact time is controlled, and (b) shows the A-A sectional view of the heat-conduction member of (a) for the block diagram of the very-low-temperature sample cooling system which made temperature control and temperature compensation possible. These Figs. are the example of drawing 5 , and another example

which made the object the same. In order to carry out setting-out control of the temperature of the cooled sample 1 or the sample electrode holder 2 to an elevated-temperature side at the temperature of arbitration In this example, a piezo-electric element 32 is formed in the division section of the first heat-conduction member 31. A heater 25 and a thermo sensor 28 are formed in the first cooling member 3 in which the sample 1 or the sample electrode holder 2 was furthermore carried. Setting-out control of the division section heat-conduction face-to-face heat contact time of the first heat-conduction member 31 is carried out at the temperature of arbitration by controlling a temperature controller 29 and the piezo-electric element driving gear 30 by the closed loop, and temperature is compensated. It is necessary to take a driving means which is not depended on a piezo-electric element 30 and by which a heat input is not made the first heat-conduction member 31 like [in other driving means] the example of drawing 5 .
[0016] Although the example explained above, the heat-conduction member constituted approximately cylindrical [an example] needs to take the means which took heat conduction into consideration enough, when taking means to divide on a fabrication, to manufacture and to combine with one.
[0017]
[Effect of the Invention] While raising the cooling attainment temperature of a very-low-temperature sample cooling system the above result, setting-out control can be carried out and the temperature of the arbitration by the side of an elevated temperature can be compensated for temperature. Furthermore, the consumption of a cooling refrigerant, especially the consumption of liquid helium small the latent heat of vaporization and expensive can be reduced, operation cost can be reduced, and improvement in the sample cooling operating time can be aimed at.

[Translation done.]